



August 12, 2016

Tom Moe USS Corporation P.O. Box 417 8771 Park Ridge Dr Mountain Iron, MN 55768

RE: Project: Toxicity

Pace Project No.: 1271183

Dear Tom Moe:

Enclosed are the analytical results for sample(s) received by the laboratory on July 25, 2016. The results relate only to the samples included in this report. Results reported herein conform to the most current TNI standards and the laboratory's Quality Assurance Manual, where applicable, unless otherwise noted in the body of the report.

If you have any questions concerning this report, please feel free to contact me.

Sincerely,

Laura Flood for Dan J Toms

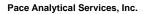
dan.toms@pacelabs.com

Law M. Sal

Project Manager

Enclosures





315 Chestnut Street Virginia, MN 55792 (218) 742-1042

CERTIFICATIONS

Project: Toxicity Pace Project No.: 1271183

Duluth Minnesota Cerification ID's

4730 Oneota St., Duluth, MN 55807 Wisconsin DNR Certification #: 999446800

Minnesota Dept of Health Certification #: 027-137-152 North Dakota Certification #: R-105

315 Chestnut Street Virginia, MN 55792 (218) 742-1042



SAMPLE SUMMARY

Project: Toxicity
Pace Project No.: 1271183

Lab ID	Sample ID	Matrix	Date Collected	Date Received
1271183001	WS006 / WS007	Water	07/25/16 11:50	07/25/16 14:45
1271183002	SW002	Water	07/25/16 12:30	07/25/16 14:45



SAMPLE ANALYTE COUNT

Project: Toxicity
Pace Project No.: 1271183

Lab ID	Sample ID	Method	Analysts	Analytes Reported	Laboratory
1271183001	WS006 / WS007	EPA 120.1	AXP	1	PASI-DUL
		SM 2320B	KJD	1	PASI-DUL
		SM 4500-CL E	APR	1	PASI-DUL
		SM 4500-H+B	CLC	1	PASI-DUL
		SM 4500-NH3 D	AXP	1	PASI-DUL
		USGS I-1338	KJD	1	PASI-DUL
1271183002	SW002	EPA 120.1	AXP	1	PASI-DUL
		SM 2320B	KJD	1	PASI-DUL
		SM 4500-CL E	APR	1	PASI-DUL
		SM 4500-H+B	CLC	1	PASI-DUL
		SM 4500-NH3 D	AXP	1	PASI-DUL
		USGS I-1338	KJD	1	PASI-DUL



ANALYTICAL RESULTS

Project: Toxicity
Pace Project No.: 1271183

Date: 08/12/2016 02:34 PM

Sample: WS006 / WS007	Lab ID: 12	71183001	Collected: 07/25/1	16 11:50	Received: 0)7/25/16 14:45 I	Matrix: Water	
Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
120.1 Specific Conductance	Analytical Me	thod: EPA 120.	1					
Specific Conductance	2010	umhos/cm	1.0	1		08/01/16 16:46	;	
2320B Alkalinity	Analytical Me	thod: SM 2320E	3					
Alkalinity, Total as CaCO3	190	mg/L	20.0	1		07/29/16 15:28	1	
4500CL E Chlorine, Residual	Analytical Me	thod: SM 4500-	·CL E					
Chlorine, Total Residual	ND	mg/L	0.020	1		07/26/16 19:37	7782-50-5	H6,N2
4500H+ pH, Electrometric	Analytical Me	thod: SM 4500-	·H+B					
pH at 25 Degrees C	8.2	Std. Units	0.10	1		07/26/16 18:50)	H6
4500 Ammonia Water	Analytical Me	thod: SM 4500-	NH3 D					
Nitrogen, Ammonia	1.6	mg/L	0.20	1		08/01/16 11:48	7664-41-7	
USGS Hardness, Total as CaCO3	Analytical Me	thod: USGS I-1	338					
Total Hardness	992	mg/L	5.0	1		07/30/16 14:26	i	
Sample: SW002	Lab ID: 12	71183002	Collected: 07/25/1	16 12:30	Received: 0)7/25/16 14:45	Matrix: Water	
Parameters	Results	Units	Report Limit	DF	Prepared	Analyzed	CAS No.	Qual
120.1 Specific Conductance	Analytical Me	thod: EPA 120.	1					
Specific Conductance	96.8	umhos/cm	1.0	1		08/01/16 16:47	•	
2320B Alkalinity	Analytical Me	thod: SM 2320E	3					
Alkalinity, Total as CaCO3	42.4	mg/L	20.0	1		07/29/16 15:30)	
4500CL E Chlorine, Residual	Analytical Me	thod: SM 4500-	·CL E					
Chlorine, Total Residual	ND	mg/L	0.020	1		07/26/16 19:41	7782-50-5	H6,N2
4500H+ pH, Electrometric	Analytical Me	thod: SM 4500-	·H+B					
pH at 25 Degrees C	6.8	Std. Units	0.10	1		07/26/16 18:52	!	H6
4500 Ammonia Water	Analytical Me	thod: SM 4500-	NH3 D					
Nitrogen, Ammonia	ND	mg/L	0.20	1		08/01/16 11:53	7664-41-7	
USGS Hardness, Total as CaCO3	Analytical Me	thod: USGS I-1	338					
Total Hardness	56.0	mg/L	5.0	1		07/30/16 14:33	}	
		-						

REPORT OF LABORATORY ANALYSIS

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QUALITY CONTROL DATA

EPA 120.1

Project: Toxicity
Pace Project No.: 1271183

QC Batch: 89661 Analysis Method:

QC Batch Method: EPA 120.1 Analysis Description: 120.1 Specific Conductance

Associated Lab Samples: 1271183001, 1271183002

METHOD BLANK: 352738 Matrix: Water

Associated Lab Samples: 1271183001, 1271183002

Blank Reporting
Parameter Units Result Limit Analyzed Qualifiers

Specific Conductance umhos/cm ND 1.0 08/01/16 16:42

LABORATORY CONTROL SAMPLE: 352737

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Specific Conductance umhos/cm 1410 1380 97 90-110

SAMPLE DUPLICATE: 352739

Date: 08/12/2016 02:34 PM

1271282001 Dup Max **RPD RPD** Parameter Units Result Result Qualifiers 335 335 0 10 Specific Conductance umhos/cm

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



Toxicity

1271183

Project:

Pace Project No.:

Alkalinity, Total as CaCO3

Date: 08/12/2016 02:34 PM

QUALITY CONTROL DATA

QC Batch: 89240 Analysis Method: SM 2320B QC Batch Method: SM 2320B Analysis Description: 2320B Alkalinity Associated Lab Samples: 1271183001, 1271183002 METHOD BLANK: 351002 Matrix: Water Associated Lab Samples: 1271183001, 1271183002 Blank Reporting Limit Parameter Result Analyzed Qualifiers Units Alkalinity, Total as CaCO3 ND 20.0 07/29/16 13:45 mg/L

LABORATORY CONTROL SAMPLE: 351001 Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Alkalinity, Total as CaCO3 mg/L 50 51.8 104 90-110 SAMPLE DUPLICATE: 351003 1271133001 Dup Max **RPD RPD** Parameter Units Result Result Qualifiers 62.1 0 10 Alkalinity, Total as CaCO3 62.1 mg/L SAMPLE DUPLICATE: 351004 1270630004 Dup Max RPD RPD Parameter Units Result Result Qualifiers 308

302

2

10

mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

Project: Toxicity Pace Project No.: 1271183

QC Batch: 89174

QC Batch Method: SM 4500-CL E

Associated Lab Samples: 1271183001, 1271183002

350566 METHOD BLANK:

Associated Lab Samples: 1271183001, 1271183002

Parameter Units Blank Reporting

Result

Matrix: Water

Analysis Method:

Analysis Description:

Limit Analyzed

SM 4500-CL E

4500CL E Chlorine, Total Residual

Qualifiers

80-120 H6,N2

Chlorine, Total Residual ND 0.020 07/26/16 19:25 H6,N2 mg/L

LABORATORY CONTROL SAMPLE: 350565

Spike Units Conc.

LCS Result

LCS % Rec % Rec Limits

Qualifiers

Chlorine, Total Residual mg/L

Parameter

Date: 08/12/2016 02:34 PM

SAMPLE DUPLICATE: 350567

1271183001 Parameter Units Result

Dup Result

0.089

RPD

89

Max **RPD**

Qualifiers

Chlorine, Total Residual ND ND 20 H6,N2 mg/L

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

Project:

Toxicity

Pace Project No.:

1271183

QC Batch: QC Batch Method: 89206

SM 4500-H+B

Analysis Method:

SM 4500-H+B

350776

Analysis Description:

4500H+B pH Electrometric

Associated Lab Samples:

1271183001, 1271183002

LABORATORY CONTROL SAMPLE:

Parameter

Spike

LCS

LCS

% Rec

pH at 25 Degrees C

Units Std. Units Conc. 7 Result

% Rec 101 Limits 98-102 H6

Qualifiers

SAMPLE DUPLICATE: 350777

Date: 08/12/2016 02:34 PM

Parameter

1271183001 Result

Dup Result

7.1

RPD

Max RPD

Qualifiers

pH at 25 Degrees C

Units Std. Units

8.2

8.1

0

10 H6

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



Toxicity

1271183

MATRIX SPIKE & MATRIX SPIKE DUPLICATE:

Parameter

Date: 08/12/2016 02:34 PM

Project:

QUALITY CONTROL DATA

Pace Project No.: QC Batch: 89591 Analysis Method: SM 4500-NH3 D QC Batch Method: SM 4500-NH3 D Analysis Description: 4500 Ammonia Associated Lab Samples: 1271183001, 1271183002 352447 METHOD BLANK: Matrix: Water Associated Lab Samples: 1271183001, 1271183002 Blank Reporting Parameter Limit Qualifiers Units Result Analyzed Nitrogen, Ammonia ND 0.20 08/01/16 11:33 mg/L LABORATORY CONTROL SAMPLE: 352446 Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers Nitrogen, Ammonia mg/L 2 1.9 96 90-110

Nitrogen, Ammonia	mg/L	ND	2	2	2.0	2.0	96	98	90-110	2	10	
MATRIX SPIKE & MATRIX SPIR	KE DUPLIC	CATE: 35245	0		352451							
			MS	MSD								
		1271326002	Spike	Spike	MS	MSD	MS	MSD	% Rec		Max	
Parameter	Units	Result	Conc.	Conc.	Result	Result	% Rec	% Rec	Limits	RPD	RPD	Qual
Nitrogen, Ammonia	mg/L	ND	2	2	2.0	2.1	97	101	90-110	4	10	

MSD

Spike

Conc.

352449

MS

Result

MSD

Result

MS

% Rec

MSD

% Rec

% Rec

Limits

Max

Qual

RPD RPD

352448

1271264001

Result

Units

MS

Spike

Conc.

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALITY CONTROL DATA

Project: Toxicity
Pace Project No.: 1271183

Date: 08/12/2016 02:34 PM

QC Batch: 89538 Analysis Method: USGS I-1338

QC Batch Method: USGS I-1338 Analysis Description: USGS T Hardness as CaCO3

Associated Lab Samples: 1271183001, 1271183002

METHOD BLANK: 352287 Matrix: Water

Associated Lab Samples: 1271183001, 1271183002

Blank Reporting
Parameter Units Result Limit Analyzed Qualifiers

Total Hardness mg/L ND 5.0 07/30/16 12:39

LABORATORY CONTROL SAMPLE: 352288

Spike LCS LCS % Rec Parameter Units Conc. Result % Rec Limits Qualifiers **Total Hardness** mg/L 100 100 100 90-110

MATRIX SPIKE & MATRIX SPIKE DUPLICATE: 352291 352292

MS MSD 1271133001 Spike Spike MS MSD MS MSD % Rec Max Parameter Units Result Conc. Conc. Result Result % Rec % Rec Limits RPD RPD Qual **Total Hardness** 100 mg/L 90.0 100 190 191 100 101 90-110 10

Results presented on this page are in the units indicated by the "Units" column except where an alternate unit is presented to the right of the result.



QUALIFIERS

Project: Toxicity
Pace Project No.: 1271183

DEFINITIONS

DF - Dilution Factor, if reported, represents the factor applied to the reported data due to dilution of the sample aliquot.

ND - Not Detected at or above adjusted reporting limit.

J - Estimated concentration above the adjusted method detection limit and below the adjusted reporting limit.

MDL - Adjusted Method Detection Limit.

PQL - Practical Quantitation Limit.

RL - Reporting Limit.

S - Surrogate

1,2-Diphenylhydrazine decomposes to and cannot be separated from Azobenzene using Method 8270. The result for each analyte is a combined concentration.

Consistent with EPA guidelines, unrounded data are displayed and have been used to calculate % recovery and RPD values.

LCS(D) - Laboratory Control Sample (Duplicate)

MS(D) - Matrix Spike (Duplicate)

DUP - Sample Duplicate

RPD - Relative Percent Difference

NC - Not Calculable.

SG - Silica Gel - Clean-Up

U - Indicates the compound was analyzed for, but not detected.

N-Nitrosodiphenylamine decomposes and cannot be separated from Diphenylamine using Method 8270. The result reported for each analyte is a combined concentration.

Pace Analytical is TNI accredited. Contact your Pace PM for the current list of accredited analytes.

TNI - The NELAC Institute.

LABORATORIES

PASI-DUL Pace Analytical Services - Duluth

ANALYTE QUALIFIERS

Date: 08/12/2016 02:34 PM

H6 Analysis initiated outside of the 15 minute EPA required holding time.

N2 The lab does not hold TNI accreditation for this parameter.



QUALITY CONTROL DATA CROSS REFERENCE TABLE

Project: Toxicity
Pace Project No.: 1271183

Date: 08/12/2016 02:34 PM

Lab ID	Sample ID	QC Batch Method	QC Batch	Analytical Method	Analytical Batch
1271183001	WS006 / WS007	EPA 120.1	89661		
1271183002	SW002	EPA 120.1	89661		
1271183001	WS006 / WS007	SM 2320B	89240		
1271183002	SW002	SM 2320B	89240		
1271183001	WS006 / WS007	SM 4500-CL E	89174		
1271183002	SW002	SM 4500-CL E	89174		
1271183001	WS006 / WS007	SM 4500-H+B	89206		
1271183002	SW002	SM 4500-H+B	89206		
1271183001	WS006 / WS007	SM 4500-NH3 D	89591		
1271183002	SW002	SM 4500-NH3 D	89591		
1271183001	WS006 / WS007	USGS I-1338	89538		
1271183002	SW002	USGS I-1338	89538		

PM: DJT

Due Date: 08/15/36 ORP

CHAIN-OF-CUSTODY / Analytical Request Docui CLIENT: USS CORP The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be c

			ADDITIONAL COMMENTS	2	70	9		9	3 SW002	1 WS001 1/ SODG /WS0		SAMPLE ID Sollsolid One Character per box. (A-Z, 0-91, -) Sample lds must be unique Sample lds must be unique Other		NATE V	Requested Due Date/TAT:	Phone: 218.749.7485 Fax:218.749.7360	Email To:	Mountain Iron, Minnesota 55768	Address: 8771 Park Ridge Dr	Company: U.S. Steel-Minntac	Section A Required Client Information:
SAMPLER NAME AND SIGNATURE PRINT Name of SAMPLER. SIGNATURE of SAMPLER.		m/se/ 251/30/400	RELINQUISHED BY / AFFE INTION DATE						0 42/ 01/21/ 5m	07 MG 7/1-1/1 1/50	ORIC LIME DATE LIME	MATRIX CODE (see SAMPLE TYPE (G=CODATE TIME DATE TIME	Valid code	s to left) COMP) COLLECTED	Container Order Number:	Client Project ID:	Purchase Order No.			Report To: Tom Moe	Section B Required Project Information:
AMORE WAS A. MOE DATE Signed:		6/300 Kustema Polson	TIME ACCEPTED BY AFFILIATION						4 2 1 1 X X X X	4 2 1 1 X X X X	# H H C H	SAMPLE TEMP AT CO		Preservatives	Pace Profile #:	Pace Project Manager	Pace Quote Reference:	Address:	Company Name:	Attention:	Section C Invoice Information:
TEMP in C Received on Ice (Y/N) Custody Sealed Cooler (Y/N) Samples Intact (Y/N)		1/as/16/1445 4 N Y	DATE TIME SAMPLE CONDITIONS						x x 002	× × × OO -	F	Ammonia Total Residual Chlori M Acute Residual Chlorine (Y		at January 515; in Issue (A), (A) My		State / Location		Regulatory Agency			Page: 1 Of 1

ace Analytical "

Document Name:

Sample Condition Upon Receipt Form

Document No.: F-DUL-C-001-Rev.01 Document Revised: 22Jan2016

Page 1 of 1

Issuing Authority:

Pace Virginia, Minnesota Quality Office

WSOOK | WSOO7 Client Name:

Sample Condition

Project #:

		MOH · IZ/IIO
SPS	Client	
ther		1071183

	nntac			MOTI - IZ. / I I O
Courier: ☐ Fed Ex ☐ UPS ☐ Commercial 전 Pace	☐USPS ☐Other:_	□сі	lient	1271183
cking Number:				
cody Seal on Cooler/Box Present? Yes	No	Seals In	tact?	Yes No Optional: Proj. Due Date: Proj. Name:
king Material: Bubble Wrap Bubble	Bags No	ne 🗌	Other:	Temp Blank? █ yes □ No
nometer Used: 📈 B00051	Type of I		Wet Γ	Blue None Samples on ice, cooling process has be
<u> </u>	p Corrected °(Biological Tissue Frozen? Yes No
o should be above freezing to 6°C Correction F				Initials of Person Examining Contents:
ain of Custody Present?	¥₹Yes	□No	□N/A	1.
ain of Custody Filled Out?	Yes	∏No	□n/a	2.
nain of Custody Relinquished?	Yes	□No	□n/a	3.
impler Name and Signature on COC?	-K Yes	□No	□n/A	4.
amples Arrived within Hold Time?	¥es	□No	□n/a	5.
nort Hold Time Analysis (<72 hr)?	¥⊋Yes	□No	□n/a	6. PH, rescl
ush Turn Around Time Requested?	□Yes	[∑No	□n/a	7.
ufficient Volume?	¥dyes	□No	□N/A	8.
orrect Containers Used?	₩ ∀ es	□No	□n/a	9.
-Pace Containers Used?	∑ Yes	□No	□n/A	·
ontainers Intact?	∑oyes	□No	 □N/A	10.
iltered Volume Received for Dissolved Tests?	□Yes	□No	S Złty/A	11. Note if sediment is visible in the dissolved containers.
ample Labels Match COC?	₹QYes	□No	□n/a	12.
-Includes Date/Time/ID/Analysis Matrix: W		_		
Il containers needing acid/base preservation will be hecked and documented in the pH logbook.		□No	□n/a	See pH log for results and additional preservati documentation
leadspace in Methyl Mercury Container	□Yes	□No	II N/A	13.
leadspace in VOA Vials (>6mm)?	 Yes	□No	□N/A	14.
rip Blank Present?	□Yes	□No	□N/A	15.
rip Blank Custody Seals Present?	 ∐Yes	. No	□N/A	
TIP Blatik Custody Seals I reserve:		•	ı	

FECAL WAIVER ON FILE

TEMPERATURE WAIVER ON FILE

Project Manager Review: At for WH Date: 7-26 L

Note: Whenever there is a discrepancy affecting North Carolina compliance samples, a copy of this form will be sent to the North Carolina DEHNR Certification Office (i.e out of

hold, incorrect preservative, out of temp, incorrect containers)

Document Name:

Sample Condition Upon Receipt Form

Document No.:

Document Revised: 22Jan2016 Page 1 of 1

Issuing Authority:

	F-1	DUL-C-0	01-Rev.01	Pace Virginia, Minnesota Quality Office
Sample Condition Upon Receipt US Steel - Min			Project #	WO#:1271183
Courier: Fed Ex UPS Commercial Pace	USPS Other:	c	Client	PM: DJT Due Date: 08/15/16 CLIENT: USS CORP
Tracking Number:				
Custody Seal on Cooler/Box Present? Yes	No	Seals In	ntact?	Yes Optional: Proj. Due Date: Proj. Name:
Packing Material: Bubble Wrap Bubble	Bags XNo	ne [Other:	Temp Blank? ☐Yes ☐ No
hermometer Used: 🔀 B00051	Type of lo	:e: 🔀	Wet [Blue None Samples on ice, cooling process has begu
Cooler Temp Read °C: 19.5 Cooler Temp remp should be above freezing to 6°C Correction F	p Corrected °C actor: <u>-0.8</u>	الا <u>د</u> الا	Date and	Biological Tissue Frozen? Yes No No No Initials of Person Examining Contents: 7125(10 kp)
Chain of Custody Present?	¥Yes	□No	□N/A	1.
Chain of Custody Filled Out?	∑ Yes	□No	□n/a	2.
Chain of Custody Relinquished?	¥Yes	□No	□n/A	3.
Sampler Name and Signature on COC?	- ₹ Yes	□No	□N/A	4.
Samples Arrived within Hold Time?	¥yes	□No	□n/a	5.
Short Hold Time Analysis (<72 hr)?	Yes	□No	□n/a	6. pH, resce
Rush Turn Around Time Requested?	□Yes	∑ No	□N/A	7.
Sufficient Volume?	¥€Yes	□No	N/A	8.
Correct Containers Used?	¥⊄Yes	□No	□n/a	9.
-Pace Containers Used?	yes	□No	□N/A	
Containers Intact?	∑oyes	□No	□n/A	10.
Filtered Volume Received for Dissolved Tests?	□Yes	□No	S≥ N/A	11. Note if sediment is visible in the dissolved containers.
Sample Labels Match COC?	Yes	□No	□N/A	12.
-Includes Date/Time/ID/Analysis Matrix:				
All containers needing acid/base preservation will be checked and documented in the pH logbook.		∏No	□n/a	See pH log for results and additional preservation documentation
Headspace in Methyl Mercury Container	□Yes	□No	∏ N/A	13.
Headspace in VOA Vials (>6mm)?	∐Yes	□No	[]N/A	14.
Trip Blank Present?	□Yes	□No	□ N/A	15.
Trip Blank Custody Seals Present?	□Yes	□No	D N/A	
Pace Trip Blank Lot # (if purchased):			•	<u> </u>
CLIENT NOTIFICATION/RESOLUTION				Field Data Required? Yes No
				Date/Time:
Comments/Resolution:				

FECAL WAIVER ON FILE

TEMPERATURE WAIVER ON FILE

7-26-14 Date: Project Manager Review: